RESEARCH ARTICLE

Green Synthesis and Characterization of Zinc Oxide Nanoparticles using Ocimum Tenuiflorum

P.Yasotha, V.Kalaiselvi*, N.Vidhya, V.Ramya
Department of Physics, Navarasam Arts and Science College for women,
Erode, Tamilnadu, India.

ABSTRACT: The present study reports the synthesis and characterization of ZnO nanoparticles by green synthesis method. The capping agent of Ocimum tnuiflorum leaf extract was used as a reducing agent in the preparation of ZnO nanoparticles. The synthesized ZnO nanoparticles were characterized by X-Ray diffraction (XRD), Fourier Transform Infrared Spectroscopy (FT-IR), scanning electron microscope (SEM) and Energy Dispersive Analysis of X–rays (EDAX). The obtained results reveal that the crystalline size, morphology and composition match well with the standard values and will be useful for antibacterial applications.

KEYWORDS: Zinc Oxide, Ocimum tenuiflorum, Morphology, Composition, Crystalline size.

https://doi.org/10.29294/IJASE.7.1.2020.1584-1588

1. INTRODUCTION

The manipulation of matter with at least one dimension sized from 1 to 100 nm is measured in Nanotechnology [1]. Nanotechnology literally means any technology on a nano scale that has applications in the real world. Nanotechnology encompasses the production and application of physical, chemical, and biological systems at scales ranging from individual atoms or molecules to submicron dimensions, as well as the integration of the resulting nanostructures into larger systems. Nanotechnology is likely to have a profound impact on our economy and society in the early 21st century, comparable to that of semiconductor technology, information technology medical sciences or cellular and molecular biology. Science and technology research in nanotechnology promises breakthroughs in areas such as material manufacturing, nano electronics, pharmaceutical, energy, biotechnology and information technology. It is widely felt that nanotechnology will be the next Industrial Revolution [2,3]. Green synthesis of nanoparticles represents an advance over other methods because it is simple, cost-effective, and relatively reproducible, and often results in more stable materials. In the method of green synthesis, there is no requirement for high pressure, energy, temperature, or toxic chemicals [4,5]. Hence, nowadays, many researchers are diverting themselves from using synthetic methods. Plants produce more stable nanoparticles compared to other means and it is very straightforward to scale up. The risk of contamination is much lower. Hence, an alternative option for synthesizing nanoparticles is by applying plants and their parts. Hence due to these advantages, green synthesis is gaining extreme importance in all the fields focusing on a greener environment. Extensive research work is carried out using plants and their parts for nanoparticles due to the ease in scaling up for larger production, apart from being cost effective and environmental friendly [6,7]. Zinc oxide is an inorganic compound used in a number of manufacturing processes. It can be found in rubbers, plastics, ceramics, glass, cement, lubricants, paints, ointments, adhesives, sealants, pigments, foods, batteries, ferrites, fire retardants, and first-aid tapes. It occurs naturally as the mineral zincates, but most zinc oxide is produced synthetically [8,9]. The synthesis of ZnO nanoparticles was reported by several research workers including in the applications of solar cell and biological sciences. This aspect necessitated their usage as antibacterial agents, noxious to microorganisms and hold good biocompatibility to human cells. Synthesis of nanoparticles can be performed using a number of routinely used chemical methods such as chemical precipitation, sonochemical, solvothermal, sol-gel process, hydrothermal decomposition and so on. The biological method of the synthesis of ZnO nanoparticles is gaining importance due to its simplicity, eco-friendliness and extensive antimicrobial activity [3,10]. Zinc Acetate Dehydrate is a moderately water-soluble crystalline Zinc source that decomposes to Zinc oxide on heating. It is readily available in most volumes, including bulk quantities. Acetates are excellent precursors for production of ultra high

**Corresponding Author: nk.arthi.kalai@gmail.com*
Received: 18.06.2020 **Accepted:** 25.07.2020 **Published on**: 25.08.2020

purity compounds, catalysts, and nano scale materials [11].

Ocimum sanctum L. (also known as *Ocimum tenuiflorum*, Tulsi) has been used as medicine for more than thousands of years in Indian traditional medicine, Ayurveda and its allied verbalism disciplines for its diverse healing properties. The plant is considered sacred and is worshipped in a sanctorum of its own in traditional Hindu temples, sacred groves, and households throughout the subcontinent and therefore its taxonomical synonym *O. sanctum* L. is more popular in Indian scientific literature [12]. It relieves people of stress, restore and improve body immunity and digestion. The chemical constituents of *Ocimum tenuiflorum* are linalool, alkaloids, ursolic acid, glycosides, carvacrol, tannins, rosmarinic acid, aromatic compound [12]. In the present study, Zinc oxide nanoparticles are synthesized by green synthesis method using *Ocimum tenuiflorum* leaf extract.

Fig-1 *Ocimum tenuiflorum* leaves

2. MATERIALS AND METHODS

2.1 MATERIAL

The chemicals such as Zinc acetate, NaOH, Acetone and distilled water utilized in this work were purchased from the company of scientific and chemicals, Erode. The *Ocimum tenuiflorum* leaves were collected from Sivagiri, Erode, Tamilnadu. India. The chemicals used in the present study are analytic grade and used as such without any further purifications.

2.2METHODS

2.2.1 Preparation of Zinc Oxide Nanoparticles

About 10.975g of Zinc acetate dehydrate powder was mixed with 100ml distilled water. The mixture was stirred 30 minutes using magnetic stirrer. During this process, the sodium hydroxide (NaOH) solution was added drop-by-drop to maintain the pH as 12. Then the solution was stirred for 30 minutes and the prepared mixture was aged for 24 hrs at room temperature. Finally the white color precipitate was obtained. It was dried at microwave oven for 30 minutes the prepared powder was kept into furnace at 400 °C for 4hrs. Then the dried sample was grained by mortar and then finally Zinc Oxide Nano particles were obtained as fine powder [13].

2.2.2 Preparation of Ocimum Tenuiflorum Leaf Extract

Plant extract was prepared by taking 10g of *Ocimum tenuiflorum* leaves. The leaves were washed several times using distilled water and boiled 15 minutes in 50ml of distilled water. Then the solution is changed to reddish color. The extract was filtered and stored at room temperature.

2.2.3 Synthesis of Leaf Capped ZnO Nanoparticles (K-ZnO)

To synthesis ZnO nano particles, 10ml of leaves extract and 10.975g of Zinc acetate dihydrate was taken into separate beakers with 100ml of distilled water. The leaf extract was added into Zinc acetate dihydrate solution and this mixture was stirred for 30minutes by using magnetic stirrer. The color of solution was changed from green to yellow color then the 5g of sodium hydroxide solution was added drop-by-drop into the mixture to maintain pH level at12 .After the 30 minutes of progression, the color of solution slowly changed into pale yellow. The synthesized sample was aged for 24 hours. Thus the settled precipitate was kept in microwave oven at 350w for 25 minutes .The dried sample was grained in a mortar and then the fine leaf capped ZnO nanoparticles were obtained [13].

2.3 Characterization Methods

2.3.1 FT-IR Spectroscopy

To identify the functional groups present in the prepared samples were studied using Fourier transform spectroscopy analysis. The IR spectrum was recorded in the range of 4000-400 cm^{-1}. FT-IR spectroscopy is widely used in industrial research for quality control and dynamic measurement. This method was also applied for cancer screening [14].

2.3.2. X-ray powder diffraction (XRD)

The prepared samples were analyzed using XRD (X-ray Diffraction) technique. This XRD pattern predicts the lattice parameter (a&c), unit cell volume and crystalline size of the sample. The XRD pattern of the prepared samples was well matched with JCPDS card no: 36-1451 (which is corresponding to hexagonal

Warzite phase). The lattice parameter of the sample was calculated using the following equation:

$$1/d2=(4(h2+hk+k2)/3a2)+(l2/c2))$$

Where, d is the spacing between the planes, a and c are the lattice parameter. Te unit cell Volume (V) of the sample was described using the given equation:

$$V = (\sqrt{3}/2) + a2+c2$$

Te average crystalline size of the sample was determined by using the scherrer's formula.

$$D = k\lambda/\beta \cos \theta$$

Where D denotes the average crystalline size of the sample, K represents the broadening constant, **λ** denotes the wavelength of Cu K**λ** radiation source (1.54A°), **β** represents the full width at half maximum, angle of diffraction is denoted by **θ**.

2.3.3SEM&EDAX

Surface morphologies of synthesized ZnO samples were analyzed using Scanning Electron Microscopic analysis (SEM). Energy dispersive spectroscopy is used to identify the elemental composition of the sample.

3. RESULTS AND DISCUSSION

3.1 XRD ANALYSIS

The XRD pattern of prepared ZnO and K-ZnO was shown in Fig 1. The prepared sample confirms hexagonal Warzite structure and well matched with JCPDS file No: 36-1451.The broad diffraction peaks of the prepared for K-ZnO at values of 2 **θ** =36.589, 47.8024, 62.500, 68.262 and 69.300 and ZnO values are at 2**θ** =36.44, 47.71, 63.03, 68.12 and 69.29 are detected. The indexed hkl planes are (101), (102), (103), (112), and (201).The average crystalline size (D) of ZnO and K-ZnO was 18.53 and 14.64nm. Thus the average crystalline size of K-ZnO reduced when compared with ZnO due to capping of leaf extract. The unit cell volume (V), lattice parameters a & c decreased due to increase in crystalline size and were shown in Table1.

3.2 FT-IR ANALYSIS

FT-IR spectrums of the prepared ZnO samples were recognized using at a wavelength range of 400-4000 cm^{-1} is shown in Fig 2. The broad peak absorbed at 3861.49 cm^{-1} and 3930.93cm^{-1}(Alcohol) which is in contact to O-H stretching bonded. C-H stretching confirms from the absorption peak of 2802.5 $cm^{-1}$and 2800.6 cm^{-1}(Alkynes). N=O stretching from the absorption the peaks at 1408 cm^{-1}(Nitro). The peak observed at 3444.87 cm^{-1} and 3589.53cm^{-1} were due to stretching vibrations of N-H (Amine) bond. Introducing capping agent has created a minor change in the functional group analysis of the samples.

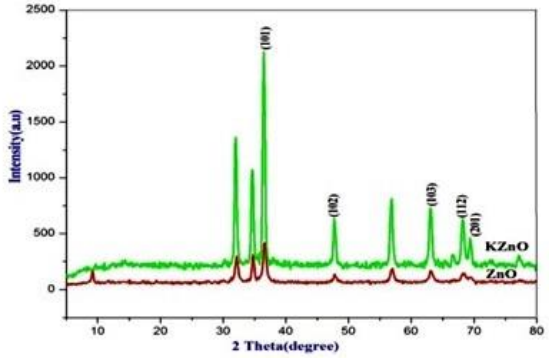


Fig-1 XRD Analysis of ZnO and K-ZnO

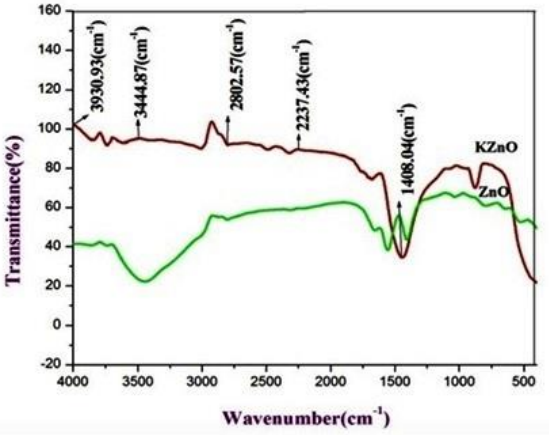


Fig-2 FT-IR Spectra of ZnO and K-ZnO

3.3 SEM AND EDAX

The micrographs of synthesized ZnO and K-ZnO nanoparticles were crystalline in nature. The synthesized pure zinc oxide nanoparticles (ZnO) predicts Needle shape structure and capped zinc oxide nano particles (K-ZnO) exhibits Non Spherical shape structure. The particle size of the ZnO was 34nm to 83nm in diameter and K-ZnO was 35 to 61nm in diameter. Energy dispersive X-ray spectroscopy (EDX) provides a qualitative and quantitative analysis. The chemical composition of the elements was determined by this technique [15]. The EDAX spectra of pure zinc oxide nano particle (ZnO) and capped zinc oxide nano particle (K-ZnO) reveal both Zinc (Zn) and Oxide (O) peaks which are observed shown in Figures 3a & 3b. EDAX spectrum also gives the intensity. Atomic weight of the elements [16] of the Sample is represented in Table (3, a, b).

Table 1 XRD results for ZnO and K-ZnO

Sample Name	2 θ (degree)	D spacing	FWHM (degree)	hkl value	Lattice Constant		Unit cell volume(v)	Crystal line size(nm)	Average crystalline size(nm)
					a=b	c			
ZnO	36.4483 47.7154 63.0386 68.1252 69.2992	2.46311 1.90448 1.47345 1.37529 1.35484	0.47030 0.49020 0.50200 0.52050 0.48490	101 102 103 112 201	3.23	5.16	46.87	17.79 17.72 18.63 18.44 20.10	18.53
K-ZnO	36.5898 47.8024 62.5000 68.2627 69.3000	2.45390 1.90122 1.48485 1.37286 1.35482	0.59320 0.56800 0.46660 0.85640 0.80000	101 102 103 112 201	3.25	5.16	47.32	14.17 15.51 11.28 20.20 12.07	14.64

Table-2 Functional groups of FT-IR

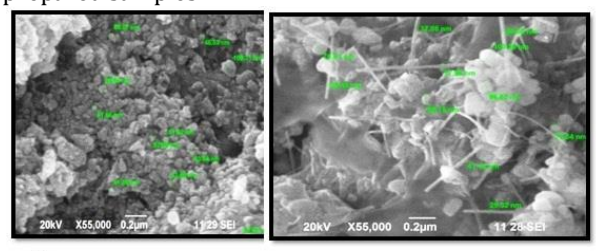
Sample Name	Wave Number (cm^{-1})				
	O-H Stretching vibration(free)	O-H Stretching vibration(bonded)	C-H Stretching vibration	N=O Stretching vibration	N-H Stretching vibration
K-ZnO	2237.43	3930.93	2802.57	1408.04	3444.87
ZnO	2318.44	3861.49	2800.64	1450.47	3589.53

Table 3 EDAX analysis of ZnO and K-ZnO nanoparticles

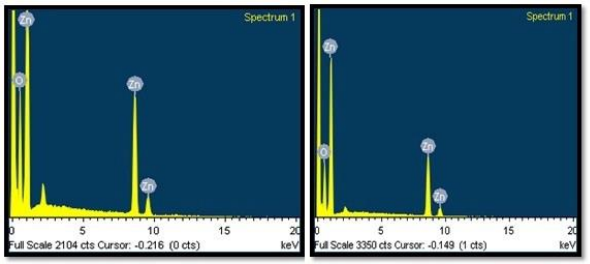
Sample	Element	App Conc.	Intensity Corrn	Weight%	Weight% Sigma	Atomic%
K-ZnO	O K	21.88	1.1241	26.42	0.69	56.46
	Zn K	50.17	0.9258	73.58	0.69	40.54
ZnO	O K	30.21	1.1484	29.05	0.59	62.59
	Zn K	50.01	0.9188	70.95	0.59	37.41

4. CONCLUSION

ZnO nanoparticles with capping agent *Ocimum tenuiflorum* leaf extract were synthesized by sol gel assisted microwave irradiation method. The synthesized samples are characterized by XRD, SEM, EDAX and FT-IR. The XRD pattern confirmed the crystalline size of the sample, lattice parameter, and unit cell volume of nanoparticles. The crystalline size decreases in K-ZnO when compared with pure ZnO. The FT-IR spectrum reveals the presence of various functional groups present in the samples and confirms the presence of Nitro and Alkynes groups. SEM analysis predicts the non spherical shaped morphological structure and Needle shape structure. EDAX analysis identified the elemental composition of the biosynthesized ZnO nano particles. Further studies are in progress to evaluate the antibacterial activity of the prepared samples.



A B



A B

Figure 3 SEM and EDAX analysis of K-ZnO (A) and ZnO (B)

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